

# Kinetics of Thermal Decomposition of Polyfunctional Substituted Azido-1,2,4-triazoles

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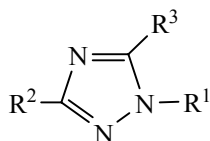
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**Abstract**—The structural and kinetics regularities and the mechanism of the limiting step of thermal decomposition of the polyfunctional substituted 1,2,4-triazoles in the melt and in solutions of inert solvents were established. The activation parameters of the limiting step were determined. Polyfunctional substituents are found to show little effect on the rate of thermal decomposition of azide group in the azole ring.

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Azido-1,2,4-triazoles are the compounds of scientific and practical interest as promising components of the rocket solid propellants [1], but their safe use is impossible without knowledge of their thermal stability, which, unfortunately, has been studied only fragmentary. Filling partially the information gap, in this study we investigated the kinetics of thermal decomposition in order to determine the effect of functional substituents on the rate and mechanism of decomposition of azido-1,2,4-triazoles.

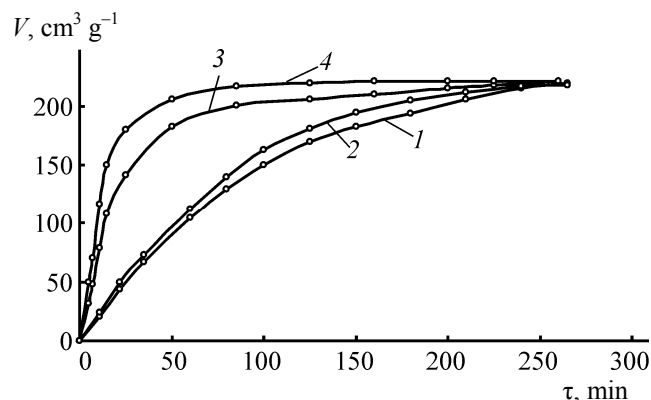


R<sup>1</sup> = H, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**I**); R<sup>1</sup> = CH<sub>2</sub>N<sub>3</sub>, R<sup>2</sup> = NO<sub>2</sub>, R<sup>3</sup> = H (**II**); R<sup>1</sup> = CH<sub>2</sub>CH<sub>2</sub>N<sub>3</sub>, R<sup>2</sup> = H, R<sup>3</sup> = H (**III**); R<sup>1</sup> = CH<sub>2</sub>CH<sub>2</sub>N<sub>3</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**IV**); R<sup>1</sup> = CH<sub>2</sub>N(NO<sub>2</sub>)CH<sub>3</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**V**); R<sup>1</sup> = CH<sub>2</sub>CH<sub>2</sub>ONO<sub>2</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**VI**); R<sup>1</sup> = CH<sub>3</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = C<sub>6</sub>H<sub>5</sub> (**VII**); R<sup>1</sup> = CH<sub>3</sub>, R<sup>2</sup> = NO<sub>2</sub>, R<sup>3</sup> = N<sub>3</sub> (**VIII**); R<sup>1</sup> = 5-NO<sub>2</sub>-tetrazol-2-yl-CH<sub>2</sub>CH<sub>2</sub>, R<sup>2</sup> = NO<sub>2</sub>, R<sup>3</sup> = H (**IX**); R<sup>1</sup> = C(NO<sub>2</sub>)(CH<sub>2</sub>N<sub>3</sub>)<sub>2</sub>, R<sup>2</sup> = NO<sub>2</sub>, R<sup>3</sup> = H (**X**); R<sup>1</sup> = CH<sub>2</sub>CCl<sub>2</sub>NO<sub>2</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**XI**); R<sup>1</sup> = C(NO<sub>2</sub>)<sub>2</sub>Br, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**XII**); R<sup>1</sup> = CCl<sub>2</sub>NO<sub>2</sub>, R<sup>2</sup> = NO<sub>2</sub>, R<sup>3</sup> = H (**XIII**); R<sup>1</sup> = CH<sub>2</sub>COOCH<sub>2</sub>C(NO<sub>2</sub>)<sub>3</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**XIV**); R<sup>1</sup> = CCl<sub>2</sub>NO<sub>2</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**XV**), R<sup>1</sup> = C(NO<sub>2</sub>)<sub>3</sub>, R<sup>2</sup> = N<sub>3</sub>, R<sup>3</sup> = H (**XVI**).

We studied the kinetics of thermal decomposition of compounds **I–XV** in the melt and as solutions in dibutyl phthalate and 1,3-dinitrobenzene. Preliminary experiments showed that the dielectric constant of

dibutyl phthalate ( $\epsilon = 6.44$ ) and 1,3-dinitrobenzene ( $\epsilon = 20.6$ ), as well as the concentration of substances in solution (1–20 wt %) do not change the reaction law and rate. At the decomposition of the melt the ratio of the substance weight to the reaction volume does not affect the rate of decomposition of compounds **II–XV** in the range of  $m/V$  from  $10^{-4}$  to  $10^{-2}$  g cm<sup>-3</sup>. The same is valid for the 2–4 times change in the ratio of the surface area of the reaction vessel to its volume,  $S/V$ . All this testifies to the absence of chain and heterogeneous processes, and points to the homogeneity of the course of the thermal decomposition reaction. Up to 40–50% conversion, the thermal decomposition is described by first-order reaction equation.

At the same time, the variation of  $S/V$  in the case of compound **I** from 1.8 to 7.4 cm<sup>-1</sup> does not affect the decomposition law in the melt (Fig. 1), but leads to 4-fold increase in the rate constant. This is probably due to the heterogeneous reactions occurring on the walls of the reaction vessel at the increase in the reaction surface area. Therefore, the study of the kinetics of thermal decomposition of **I** in the melt was carried out at  $S/V = 0.5–1.0$  cm<sup>-1</sup>, when the potential contribution of heterogeneous reactions to the overall rate of the process was very small. In addition, for compound **I** the kinetics of accumulation of molecular nitrogen during the thermal decomposition was also studied. Kinetic curves for the release of molecular nitrogen to the conversion of about 50% were practically the same as the curves of the total gas accumulation, and the values of the rate constants calculated from the curves



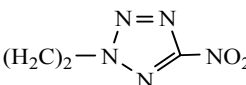
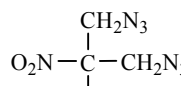
**Fig. 1.** Effect of  $S/V$  on thermal decomposition of compound **I** at 150°C,  $\text{cm}^{-1}$ : (1) 1.80, (2) 2.32, (3) 4.10, (4) 5.74.

of nitrogen evolution coincided with those found from the curves of the total gas production within the error of their determination. Kinetic parameters of thermal decomposition of the compounds studied are listed in Table 1.

The study of the gaseous products at the decomposition of some azides showed that the main product was the molecular nitrogen. In the case of compounds containing, in addition to the azido function, a nitrate, bromodinitro- or -trinitromethyl group, even at the reaction beginning or at a relatively low degree of conversion a decomposition of this group is observed, so that carbon oxides appear together with the molecular nitrogen. For example, at the decomposition of compound **V** in dichloroethane at 150°C, upon increase in the degree of conversion the intensity of the absorption band of azido group ( $2160\text{ cm}^{-1}$ ) in the IR spectrum decreases, while the intensity of the bands of  $\text{N}-\text{NO}_2$  group ( $1545, 1300\text{ cm}^{-1}$ ) remains almost constant, and the nitrogen oxides begin to appear later, as the conversion grows (18–20%). The data on this question for some azido compounds are given in Table 2.

In the IR spectrum of the condensed residue formed at the decomposition of compound **I** (Fig. 2) the azido group absorption band disappears ( $2160\text{ cm}^{-1}$ ), while

**Table 1.** Activation parameters of thermal decomposition of polyfunctional azido-1,2,4-triazoles in the melt and in solutions of dibutyl phthalate and *m*-dinitrobenzene

Comp. no.	Substituent			Conditions of decomposition	$\Delta T, ^\circ\text{C}$	$k_{160^\circ\text{C}} \times 10^5, \text{s}^{-1}$	$E_a, \text{kJ mol}^{-1}$	$\log A$	$\Delta S_{160^\circ\text{C}}^\ddagger, \text{J mol}^{-1} \text{K}^{-1}$
	$\text{R}^1$	$\text{R}^2$	$\text{R}^3$						
<b>I</b>	H	$\text{N}_3$	H	Melt	130–170	4.5	139.5	13.49	1.8
				Melt, measuring $\text{N}_2$	150–170	4.3	140.7	13.59	3.8
				Melt, $S/V=(0.5-1)\text{ cm}^{-1}$	140–170	2.0	160.1	15.61	42.4
				Dibutylphthalate solution	130–170	2.2	159.4	15.56	41.5
				<i>m</i> -Dinitrobenzene solution	150–190	0.09	162.7	14.59	22.9
<b>II</b>	$\text{CH}_2\text{N}_3$	$\text{NO}_2$	H	<i>m</i> -Dinitrobenzene solution	150–190	0.09	162.7	14.59	22.9
<b>III</b>	$\text{CH}_2\text{CH}_2\text{N}_3$	H	H	Melt	160–190	0.08	163.6	14.65	24.0
<b>IV</b>	$\text{CH}_2\text{CH}_2\text{N}_3$	$\text{N}_3$	H	<i>m</i> -Dinitrobenzene solution	140–170	2.4	156.3	15.23	35.2
<b>V</b>	$\text{CH}_2\text{N}(\text{NO}_2)\text{CH}_3$	$\text{N}_3$	H	Dibutylphthalate solution	140–170	2.1	154.4	14.92	29.2
<b>VI</b>	$\text{CH}_2\text{CH}_2\text{ONO}_2$	$\text{N}_3$	H	Melt	130–170	3.4	158.4	15.64	43.0
				Dibutylphthalate solution	130–180	2.9	158.5	15.58	41.9
<b>VII</b>	$\text{CH}_3$	$\text{N}_3$	$\text{C}_6\text{H}_5$	Melt	130–170	4.3	156.5	15.21	34.8
				<i>m</i> -Dinitrobenzene solution	130–170	4.5	158.1	15.72	44.5
<b>VIII</b>	$\text{CH}_3$	$\text{NO}_2$	$\text{N}_3$	Melt	130–170	2.6	156.2	15.26	35.7
				<i>m</i> -Dinitrobenzene solution	130–170	2.8	159.2	15.65	43.2
<b>IX</b>		$\text{NO}_2$	H	Dibutylphthalate solution	170–200	0.06	163.3	13.46	1.3
<b>X</b>		$\text{NO}_2$	H	<i>m</i> -Dinitrobenzene solution	150–180	0.5	161.7	15.18	34.2

**Table 1.** (Contd.)

Comp. no.	Substituent			Conditions of decomposition	$\Delta T$ , °C	$k_{160^\circ\text{C}} \times 10^5$ , s <sup>-1</sup>	$E_a$ , kJ mol <sup>-1</sup>	log $A$	$\Delta S_{160^\circ\text{C}}^\ddagger$ , J mol <sup>-1</sup> K <sup>-1</sup>
	R <sup>1</sup>	R <sup>2</sup>	R <sup>3</sup>						
<b>XI</b>	CH <sub>2</sub> CCl <sub>2</sub> NO <sub>2</sub>	N <sub>3</sub>	H	Melt	140–170	2.1	156.7	15.18	34.2
				Dibutylphthalate solution	140–170	1.9	157.4	15.31	36.7
<b>XII</b>	C(NO <sub>2</sub> ) <sub>2</sub> Br	N <sub>3</sub>	H	Dibutylphthalate solution	120–150	327.3	130.4	14.24	16.2
<b>XIII</b>	CCl <sub>2</sub> NO <sub>2</sub>	NO <sub>2</sub>	H	Dibutylphthalate solution	145–165	1.8	157.3	15.23	35.2
<b>XIV</b>	CH <sub>2</sub> COOCH <sub>2</sub> C(NO <sub>2</sub> ) <sub>3</sub>	N <sub>3</sub>	H	Dibutylphthalate solution	160–180	2.2	159.9	15.63	42.8
<b>XV</b>	CCl <sub>2</sub> NO <sub>2</sub>	N <sub>3</sub>	H	Dibutylphthalate solution	140–160	3.5	156.4	15.41	38.6
<b>XVI</b>	C(NO <sub>2</sub> ) <sub>3</sub> [3]	N <sub>3</sub>	H	Melt	100–140	66.0	139.7	14.67	24.4
				Dibutylphthalate solution	100–150	63.3	140.6	14.76	26.2

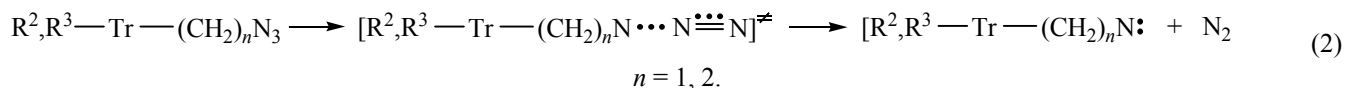
the absorption bands characteristic of the triazole ring (1535, 1460, 1380, 870, 830 cm<sup>-1</sup>) are retained. New absorption bands at 1620–1630 cm<sup>-1</sup> and at 3400–3600 cm<sup>-1</sup> appear, which can be attributed to the 3,3'-azo-1,2,4-triazole, and 3-amino-1,2,4-triazole, respectively.

The first thing that attracts attention when considering the data in Table 1 is virtually the same activation energy (within the error of its determination) for most of the polyfunctional substituted 3(5)-azido-1,2,4-triazoles, which is close to the activation energy

of phenylazide, 157.7 kJ mol<sup>-1</sup> [4]. Compound **I** (melt), **XII**, and **XIII** are the exception whose activation energy is  $E_a = 161.6$ – $163.7$  kJ mol<sup>-1</sup>. For compounds **II**, **III**, **IX**, and **X**,  $E_a = 161.7$ – $163.7$  kJ mol<sup>-1</sup>, as for the substituted alkylazides [4–6], and their thermal decomposition, as well as that of phenylazide, proceeds with the primary break of the N<sup>1</sup>–N<sup>2</sup> bond in the azido group, the decomposition of compounds **I**, **IV**–**VIII**, **XI**, **XIV**, and **XV** follows reaction (1) and of compounds **II**, **III** and **X**, reaction (2).



Tr is 1,2,4-triazol-5-yl.



Taking into account the activation energy and entropy of the thermal decomposition (Table 1) the reactions (1) and (2) should be regarded as proceeding through a semi-rigid activated complex [4, 7].

The nitrenes arising in the reactions (1) and (2) have short lifetimes and, given the composition of the condensed phase formed at the decomposition of compounds **I**, **IV**–**VIII**, **XI**, **XIV**, and **XV**, are

**Table 2.** The composition of gaseous products of thermal decomposition of azido compounds **I**, **VII**, **VIII** and **XIII** at 140°C

Comp. no.	Composition (%), under a conversion		
	5	20	100
<b>I</b>	N <sub>2</sub> (100)	N <sub>2</sub> (100)	–
<b>VI</b>	N <sub>2</sub> (100)	N <sub>2</sub> (70.7), CO, and NO (11.3), CO <sub>2</sub> (8.7), N <sub>2</sub> O (9.3)	N <sub>2</sub> (68.1), CO, and NO (11.3), CO <sub>2</sub> (13.1), N <sub>2</sub> O (12.4)
<b>VIII</b>	N <sub>2</sub> (100)	N <sub>2</sub> (100)	N <sub>2</sub> (100)
<b>XVI</b>	N <sub>2</sub> (14.5), NO (31.2), N <sub>2</sub> O (2.7), CO <sub>2</sub> (51.6)	–	N <sub>2</sub> (14.8), NO (28.4), N <sub>2</sub> O (3.5), CO <sub>2</sub> (53.3)

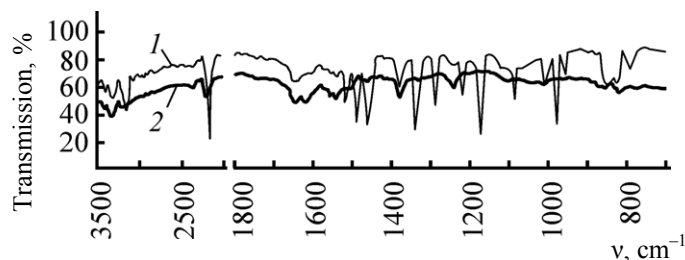
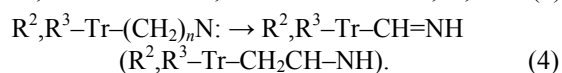
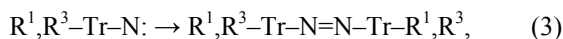


Fig. 2. IR spectrum of condensed products of thermal decomposition of compound **I** in the melt: (1) initial, (2) 80% conversion.

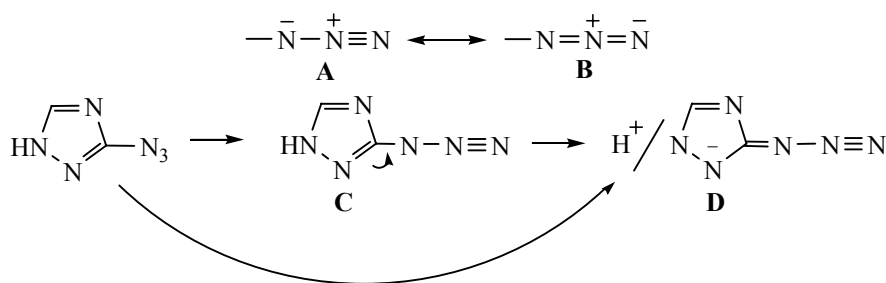
stabilized through a dimerization [reaction (3)], and in the case of compounds **II**, **III**, and **X**, through an isomerization of nitrene [reaction (4)].



The mechanisms 1–4 explain well the effect of substituents on the rate of decomposition of the azido group in the azole ring. If we take into account that the reactivity of the azido group depends not only on the strength of the broken  $N^1\text{--}N^2$  bond but also on the thermodynamic stability of the resulting electron-deficient nitrene, then the conjugation of nitrene nitrogen with the double bond should increase the thermodynamic stability of nitrene and the reactivity of the azido group of the original azole. Therefore, the electron-donating substituents in the azole ring should promote the thermal decomposition. However, Table 1 shows that both electron-donating and electron-

accepting substituents in the azole ring ( $\text{NO}_2$ ,  $\text{C}_6\text{H}_5$ ,  $\text{CH}_3$ ) have little effect on the rate of thermal decomposition of the azide group, since there is no resonance conjugation between them and the reaction centre. For this reason, the substituents  $R^1 = \text{CH}_2\text{N}(\text{NO}_2)\text{CH}_3$ ,  $\text{CH}_2\text{CH}_2\text{N}_3$ ,  $\text{CH}_2\text{CH}_2\text{ONO}_2$ ,  $\text{CH}_2\text{CCl}_2\text{NO}_2$ , and  $\text{CH}_2\text{COOCH}_2\text{C}(\text{NO}_2)_3$  virtually “do no work.”

The decrease in the activation energy of the thermal decomposition of compound **I** in the melt (first two lines of Table 1) can be attributed to the ionization of the 3-azido-1,2,4-triazole, which is a weak NH-acid [8]. The reaction of ionization of N–H bond, as noted above, is heterogeneous, it occurs on the walls of the reaction vessel, and shows a peculiar effect on the reactivity of the azido group. Thus, according to two octet resonance structures of azido group (**A** and **B**), the order of breaking  $N^1\text{--}N^2$  bond is equal to 1.5 [9]. This double-bonding in the anion is less than in the 3-azido-1,2,4-triazole molecule.

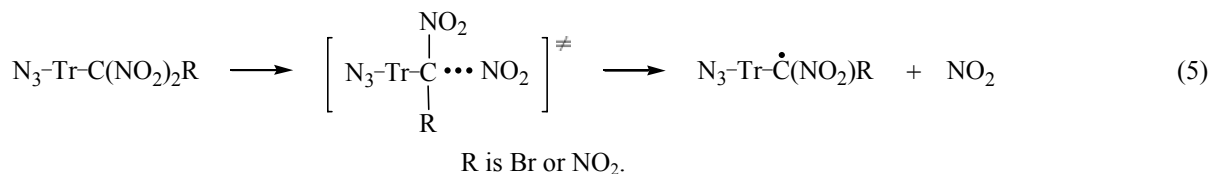


Therefore, the thermal decomposition of the ionized form of 3-azido-1,2,4-triazole **D** proceeds with lower activation energy than the non-ionized form **C**. At the same time, the ionized form **D** is a stronger electron-donating substituent compared with the molecular one and favors the decomposition.

Compared to other substituted azido-1,2,4-triazoles, the activation energy of compounds **XII**, **XVI** is lower. It may be due to the shift of the reaction site to the carbon atom of the substituted dinitromethyl group

and, consequently, to the change in the mechanism of the decomposition involving the homolytic cleavage of the C– $\text{NO}_2$  bond as the limiting step.

This is consistent with the analysis of the gas phase of the decomposition products (Table 2), as well as with the IR-spectroscopic study of the parent compound **XVI**, which showed gradual disappearance of the most intense bands at 800, 1300, and 1600  $\text{cm}^{-1}$  in the course of the  $\text{C}(\text{NO}_2)_3$  group decomposition. Note that the shifts of these bands depending on the



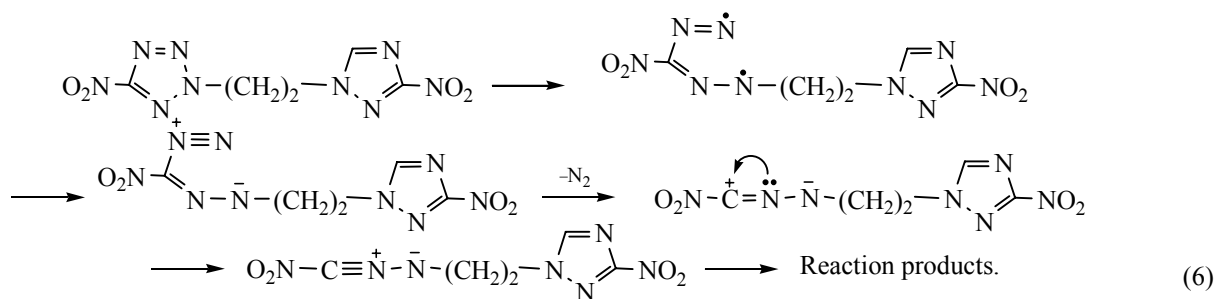
chemical environment at the trinitromethyl groups and the aggregative state are usually negligible [10], which is important for their identification. As for the azido function in the azoles, the intensity of the absorption band at 2160 cm<sup>-1</sup> (solution in methylcyclohexane) remains practically unchanged in the IR spectrum of compound **XVI** up to 10–12% conversion.

In compounds **II**, **III**, and **X** the azido group is in the alkyl fragment, so they should be regarded as alkylazides with substituted 1,2,4-triazolyl functions. Compound **IX** containing a “bent” azido group may also be assigned to this group. The presence of 3-nitroazolyl ring in the α-position to the azido group in compound **II** tenfold increases the rate of the thermal

decomposition (Table 1) compared with methylazide ( $E_a = 170.7 \text{ kJ mol}^{-1}$ ,  $\log A = 14.45$ ,  $k_{160^\circ\text{C}} = 7.0 \times 10^{-7} \text{ s}^{-1}$  [5]) due to the steric effect of substituents.

If the azole ring is in the β-position, its effect is almost unseen in the rate of thermal decomposition of compound **III**. At the same time, as in the compounds with the nitrogen–nitrogen bonds, for example, the nitramines  $(\text{TrCH}_2)_2\text{N-NO}_2$  and  $(\text{TrCH}_2\text{CH}_2)_2\text{N-NO}_2$ , the ratio of the rate constants of the thermal decomposition of α- and β-substituted derivatives at 160°C is 98 [11].

Compound **IX** decomposes homolytically, with the primary break N<sup>2</sup>–N<sup>3</sup> bond in the tetrazole ring by another route than in compound **III**.



It is noteworthy that the activation energy and the rate of thermal decomposition of the bent azido group agree well with the corresponding parameters for the linear form of azido group (compound **III**, Table 1). However, in the latter case, the activation entropy of the transition state is greater due to releasing of internal rotation of the broken N<sup>2</sup>–N<sup>3</sup> bond, which is inhibited in the ground state of the molecule, and due to a decrease in the frequency of deformation vibrations of fragments associated with this bond [4].

In contrast to compounds **III** and **IX** with β-substituents decomposing with almost equal rates, the thermal decomposition of compound **X**, having a β-branched substituent, proceeds with the rate 6.3–8.3 times higher. This increase in the rate, in our opinion, is due to the induction effect of three electron-accepting β-substituents, which, judging by the activation energy, have little effect on the strength of

the N<sup>1</sup>–N<sup>2</sup> bond, but reduce the thermodynamic stability of the formed nitrene.

Activation parameters of thermal decomposition of compounds **XII**, **XIV**, and **XVI** differ considerably, due to the different steric effect of α-substituents in the *gem*-dinitromethyl function [3]. The reduced steric influence on the *gem*-dinitromethyl group in compound **XIV** (the sum of the steric constants of the α-substituents CH<sub>2</sub>CH<sub>2</sub> and NO<sub>2</sub> Σ*E*<sub>s</sub> = –2.15) compared to **XVI** (Σ*E*<sub>s</sub> = –5.64) increases the thermal stability (the rate constant decreases ~29 times) of the *gem*-dinitromethyl group. Therefore, the thermal decomposition is initiated at the nitrogen atom N<sup>1</sup> of the azido group. Using IR spectroscopy, it was found that when the degree of conversion of compound **XIV** was 5–7% the intensity of the absorption band of azido group in the ring (2160 cm<sup>-1</sup>) decreased, and in the gas phase the only detected product was the molecular

nitrogen, therewith the most intense absorption bands of *gem*-trinitromethyl group at 800, 1300 and 1000  $\text{cm}^{-1}$  remained unchanged. Nitrogen and carbon oxides appear in the later stages of decomposition (~12–15%). Similarly, the thermal decomposition of compound **XI** occurs through the primary homolysis of the  $\text{N}^1\text{--N}^2$  bond in the azido function (Table 1).

The thermal decomposition of compound **XV** to 5% conversion proceeds with the formation of molecular nitrogen and nitrogen dioxide, and therefore we can assume that it takes two independent parallel routes, through homolysis of  $\text{C--NO}_2$  and  $\text{N}^1\text{--N}^2$  bonds, with a slight predominance of the second route. In our case, the infrared spectroscopy cannot distinguish between these ways of decomposition, but a comparison of compounds **XIII** and **XV** favors the second route.

Thus, we conclude that the rate and activation parameters of thermal decomposition of polyfunctional substituted 3(5)-azido-1,2,4-triazole are affected significantly by the resonance conjugation of azido group with the azole ring, while the nature of the substituent in the azoles has little effect when the substituent does not include a reaction centre to initiate the thermal decomposition reaction. A similar pattern of the influence of nature of substituents on the rate of thermal decomposition has been observed previously [13] for *m*-substituted phenylazides.

#### EXPERIMENTAL

The compounds **I–XVI** were synthesized and purified by known methods [2, 14], they were chromatographically pure and contained 99.4–99.8% of the basic substance.

Thermal decomposition of compounds **I–XVI** was studied by the manometric method using a Bourdon type glass gauge [15] at a residual pressure of air in the reaction volume  $10^{-2}$  to  $10^{-3}$  mm Hg. To determine the effect of  $S/V$  on the rate of decomposition of compounds in the melt we used the Bourdon's manometers with the reaction volume in the form of a globe, which allowed us to determine reliably the surface of the reaction vessel.

Along with manometry, we used volumetric method, by measuring the accumulation of molecular nitrogen. Both methods yielded the same results in the calculation of the rate constants.

Dibutyl phthalate was washed with 5 wt % sodium carbonate and water, dried with magnesium sulfate and then fractionally distilled in a vacuum:  $n_D^{20}$  1.4931

(published: 1.4926). 1,3-Dinitrobenzene was recrystallized twice from ethanol–chloroform (1:1), mp 89.5°C (published: 89.7°C).

The analysis of gaseous decomposition products was carried out on a LKhM-72 chromatograph (temperature of the column with charcoal 140°C) and IR spectrometer UR-10 with the gas cell. Spectra of condensed products of thermal decomposition were obtained in solutions of chloroform and dichloroethane.

The rate constants were calculated with the equation of the first order reaction, as well as by the Guggenheim method [16]. The error in determining the rate constants did not exceed 6.5% in both methods. The r.m.s. error of activation energy was 6.2  $\text{kJ mol}^{-1}$ , and of the logarithm of preexponential factor, 0.53 logarithmic units.

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